

Isoperibolic Calorimetric Measurements of the Fleischmann-Pons Effect

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ABSTRACT

Important advantages exist for selecting a Dewar type isoperibolic calorimeter for measurements of anomalous excess enthalpy produced by the Fleischmann-Pons Effect (FPE). These advantages include a wide dynamic range for both the cell temperature and cell input power, direct visual observations inside the cell during calorimetric experiments, relative low cost, self-purification of the system, the safety of an open system, and heat transfer mainly by electromagnetic radiation. Various generations of this calorimetry are described along with the mathematical modeling. The use of control or “blank” experiments, such as replacing palladium by platinum, show that anomalous excess power is measurable to within ± 0.1 mW using this electrochemical calorimetry. The application of this Dewar isoperibolic calorimetry at other laboratories such as NHE (Japan), Grenoble (France) and Harwell (U.K.) is discussed. Variations of isoperibolic calorimetry used by China Lake, Caltech, and M.I.T. are also examined where the main heat transfer pathway is by conduction. An improved version of the China Lake isoperibolic calorimeter is capable of measuring the small excess power (6.5 mW) produced at the beginning of an experiment by the exothermic absorption of deuterium into palladium.

Introduction: Why choose a Dewar Isoperibolic Calorimeter?

The Dewar type of isoperibolic calorimeters developed by Fleischmann and Pons provides a wide dynamic range for both the cell temperature and cell input power. The experimental temperature range can be as great as the liquid range of the electrolyte system (3.82°C to 101.42°C for pure D₂O). For typical D₂O+0.1 M LiOD electrolytes, the temperature range used is typically from 20°C dictated by the bath temperature up to the boiling point of the electrolyte. The maximum cell input power will vary with the size of the cell, but input powers up to 10 W are possible before the cell contents are driven to boiling temperatures.

The ability provided by the Dewar type cells for directly observing processes inside the cell is a very important advantage. This is especially important for co-deposition and cell boil-off experiments. Other calorimetric systems generally do not provide for visual observations inside the cell.

The relatively low cost of the Dewar type cells is another important factor for the selection of this isoperibolic system. This makes possible the simultaneous use of multiple cells involving different experiments in a single water bath of sufficient size for handling the total heat output. The major cost is simply the construction of glass Dewar cells with a high vacuum between the

inner and outer glass surfaces. The top portion (about 30%) needs to be silvered to minimize the effect of the decreasing electrolyte level caused by the electrolysis of D₂O to produce D₂ and O₂ gases. Larger cells will obviously exhibit smaller changes in the electrolyte level during electrolysis.

Another important positive feature of these Dewar cells, as well as other open systems, is that they are self-purifying. Ordinary H₂O will act as a poison to the Fleischmann-Pons effect (FPE) in D₂O+0.1 M LiOD electrolytes. Because H is preferentially electrolyzed versus D at the cathode, any H₂O contamination will be gradually removed during electrolysis. In closed systems, the initial H₂O contamination will remain trapped throughout the experiment. Not to be overlooked is the inherent safety of an open system that allows the deuterium and oxygen gases to escape as fast as they are generated.

The main heat transfer pathway for Dewar isoperibolic cells is via electromagnetic radiation (mostly infrared radiation¹). The radiative heat transfer coefficient, therefore, can be estimated by using the Stefan-Boltzmann constant ($k_B=5.6704 \times 10^{-12} \text{ W cm}^{-2} \text{ K}^{-4}$) and the experimental surface area of the inner, unsilvered portion of the glass surface of the Dewar cell. This provides a useful guide to the integrity of the Dewar vacuum and the extent of minimization of heat transfer pathways via conduction. Because of the predominate heat transfer by electromagnetic radiation, this Dewar cell system has no memory effect. For example, gas bubbles or stagnant layers that form on the inner cell wall will exert no effect on the radiative heat transfer coefficient. Any media in the cell will suffice that maintains the temperature of the inner cell wall. Furthermore, small devices for nuclear measurements can be inserted into these Dewar cells without significantly affecting the calorimetry.

The skill of the experimenter in isoperibolic calorimetry using Dewar cells is mainly exhibited in accurately determining the radiative heat transfer coefficient (k_R) and the water equivalent of the system (C_pM) consisting of the heat capacity of D₂O (C_p) and the equivalent D₂O mass (M) of the calorimetric system. For a given cell, these important parameters (k_R , C_pM) will remain largely unchanged for all experiments conducted in a similar manner in the same cell, i.e., using the same mass of D₂O+0.1 M LiOD, electrodes, thermistors, and internal heaters. These important calorimetric parameters (k_R , C_pM) can be most accurately determined in control or “blank” experiments such as using Pt/D₂O+0.1 M LiOD in place of Pd/D₂O+0.1 M LiOD [1, 2]. The use of controls avoids the all too frequent problem of attributing calorimetric errors to excess enthalpy production. The modeling of the Dewar isoperibolic calorimetric cell and the resulting mathematical equations may appear daunting, but these calculations are easily handled accurately by computers or even hand calculators as long as the k_R and C_pM parameters are properly determined.

The Fleischmann-Pons Dewar System

The Fleischmann-Pons (F-P) isoperibolic calorimetry using a Dewar type cell evolved through various designs dating back to the early 1980's. The major changes involved the dimensions

¹ From Wien's Law, the wavelength of maximum energy density, λ_{max} , is $8.82 \times 10^{-6} \text{ m}$ (8820 nm) for a cell temperature of 60°C (333.15 K). Visible light consists of much shorter wavelengths between 400 and 750 nm.

selected for the cell as well as incorporating the silvering of the top portion of the cell. Proper scaling of the system is critical because the cell diameter and length determine the volume of electrolyte used, the rate of change of the electrolyte level, the effectiveness of stirring by the electrolysis gases, the dynamic range for power input, and the magnitude of k_R and $C_p M$. These changes led to the ICARUS (Isoperibolic Calorimetry: Acquisition, Research and Utilities System) series of cells used in later experiments.

The ICARUS 1 to 3 calorimeters were for lower temperatures up to boiling, and the ICARUS 4 to 9 calorimeters allowed long-term maintenance of boiling conditions. Results of ICARUS 9 experiments have been presented [3]. The ICARUS 10 to 13 were designed and constructed for further studies of boiling, but they were never put in use [4]. A schematic diagram for the ICARUS-14 calorimeter is available, but this calorimeter was never constructed [4]. An example of the ICARUS-1 type cell used at the New Hydrogen Energy (NHE) laboratory at Sapporo, Japan from 1994 to 1998 had a filled electrolyte volume of 90 cm³, an inner diameter of 2.5 cm, an outer diameter of 4.2 cm, and a length of 25.0 cm with the top 8.0 cm silvered. A similar Fleischmann-Pons Dewar cell was used in France by Longchamp and Bonnetain [5].

The modeling and mathematical equations of the Fleischmann-Pons Dewar isoperibolic calorimeter have been previously presented [1, 2, 4, 6, 7]. These equations are given in Appendix I, and a list of symbols and units are found in Appendix II. This modeling must include all power sources that flow into and out of the calorimeter (P_{calor}). These power sources are the electrical power of the electrochemistry (P_{El}), the net electromagnetic radiation (P_R), power due to conduction (P_C), power applied by the internal heater (P_H), if any, power carried away by the electrolysis gases exiting the cell (P_{gas}), power due to the rate of any pressure-volume work done by the generated gases (P_W), and any anomalous power sources (P_X). The resulting equation is

$$P_{calor} = P_{El} + P_H + P_X + P_{gas} + P_R + P_C + P_W \quad (1)$$

where each power term is a function of time $P(t)$. As usual in thermodynamics, heat or power added to the system (calorimeter) is positive in sign while a negative sign is used for power that flows out of the calorimeter into the surroundings. The mathematical expressions for P_{calor} , P_{El} , P_{gas} , P_R , P_C and P_W are given in Appendix I. For Dewar cells where the bulk of heat transfer from the cell to the water bath is by electromagnetic radiation, the much smaller P_C and P_W terms can be incorporated into a pseudo radiative power term, P'_R , where

$$P'_R = P_R + P_C + P_W = -k'_R f(T) \quad (2)$$

and $f(T) = T_{cell}^4 - T_b^4$. The substitution of Eq. 2 into Eq. 1 yields

$$P_{calor} = C_p M (dT_{cell} / dt) = P_{El} + P_H + P_X + P_{gas} - k'_R f(T) \quad (3)$$

where P_{El} and $k'_r f(T)$ are generally the largest terms. Equation 3 is the fundamental equation used for isoperibolic calorimetry in Dewar cells. It should be noted that no valid scientific challenge has been made in the past nineteen years to the validity of any of these calorimetric equations. Mathematically, this Equation 3 is a nonlinear, inhomogeneous differential equation. For a given electrolyte level, the pseudoradiative heat transfer coefficient (k'_r) and the heat capacity of the system ($C_p M$) can be treated as constants. For large Dewar cells silvered at the top, k'_r exhibits only a weak time dependency

$$k'_r = (k'_r)^0(1 - \alpha t) \quad (4)$$

that is due to the slowly changing electrolyte level during electrolysis.

By assuming $P_X=0$, a lower bound heat transfer coefficient, $(k'_r)_1$, can be calculated by rearranging Eq. 3

$$(k'_r)_1 = (P_{El} + P_H + P_{gas} - P_{calor}) / f(T) \quad (5)$$

The use of Eq. 5 is generally the simplest starting point for the analysis of calorimetric data; therefore $(k'_r)_1$ should always be calculated at the beginning of an experimental analysis. For blank or control experiments, P_X will be very small, thus $(k'_r)_1$ will be close to the true heat transfer coefficient, $(k'_r)_2$ given by

$$(k'_r)_2 = (P_{El} + P_H + P_X + P_{gas} - P_{calor}) / f(T) \quad (6)$$

Subtracting Eq. 5 from Eq. 6 yields

$$P_X = [(k'_r)_2 - (k'_r)_1] f(T) \quad (7)$$

In any experiment there will likely be time periods where P_X is small, thus $(k'_r)_1$ will approach the value of $(k'_r)_2$.

For control experiments, a very small excess power is measurable ($1.1 \text{ mW} \pm 0.1 \text{ mW}$) that is due to the controversial recombination of the $D_2 + O_2$ electrolysis gases [1, 2]. This effect is actually due solely to the reduction of O_2 gas at the cathode because D_2 cannot be oxidized at the platinum oxide covered anode in these experiments.

It is convenient to cast Eq. 6 into a straight-line form, $y=mx+c$, to yield

$$(P_{El} + P_H + P_X + P_{gas}) / f(T) = (C_p M dT_{cell} / dt) / f(T) + (k'_r)_2 \quad (8)$$

where Eq. 3 is used for P_{calor} . The left-hand-side (L.H.S.) of Eq. 8 represents the variable y , the slope $m = C_p M$, $x = (dT_{cell}/dt)/f(T)$ and the constant $c = (k'_r)_2$.

Analogous to the differential equations of chemical kinetics, mathematical integration yields more accurate results than the direct use of the differential equation. The numerical integration of Eq. 8, however, requires the selection of time periods where the excess power, P_X , is constant with time. It is also important for integration to select time periods where there are significant changes in the cell temperature with time such as the time period where the internal cell heater power, P_H , is applied ($t_1 \leq t \leq t_2$). Previous experiments have shown that backward integration from t_2 (cessation of heater power) produces the most accurate results [1, 2, 4, 7]. This backward integration of Eq. 8 from t_2 back to time, t , yields

$$\frac{\int_{t_2}^t P_{net}(t)dt + P_x(t-t_2)}{\int_{t_2}^t f(T)dt} = \frac{C_p M [T_{cell}(t) - T_{cell}(t_2)]}{\int_{t_2}^t f(T)dt} + (k'_R)_{262} \quad (9)$$

where $P_{net}(t) = P_{EI}(t) + P_H(t) + P_{gas}(t)$.

Various types of heat transfer coefficients such as $(k'_R)_{262}$ in Eq. 9 are designated by $(k'_R)_{i,j,k}$ where $i = 1$ designates differential, $i = 2$ signifies backward integration, $i = 3$ signifies forward integration, $k = 1$ denotes “lower bound” (assumes $P_X = 0$) and $k = 2$ signifies “true”. If used, j designates the particular time period of the two-day cycle used for the integration. For example, $j = 6$ used in Eq. 9 is especially useful because this time period between t_1 and t_2 involves the application of the cell heater power and large changes in the cell temperature. Note that Eq. 9 is in the straight-line-form where $y = \text{L.H.S.}$, $m = C_p M$ and $c = (k'_R)_{262}$. It should also be noted that the extrapolation of Eq. 9 to $x = 0$ ($t = t_2$) automatically removes the effect of $C_p M$ on the values of the derived heat transfer coefficient. This is a desirable feature because the heat capacity of the system, $C_p M$, has the highest error. Furthermore, $(k'_R)_{262}$ is the value of the integral heat transfer coefficient at $t=t_2$, the mid-point of the two-day cycle (see Eq. 4). Forward integration of this same experimental data simply involves the substitution of t_1 for t_2 in Eq. 9 to yield $(k'_R)_{362}$. Similarly, assuming $P_X=0$ in Eq. 9 would yield $(k'_R)_{261}$.

The use of Eq. 9 requires an evaluation of P_X for the time period of interest. The obvious advantage of blank or control experiments is that P_X will be close to zero. For other experiments, the calculation of $(k'_R)_1$ using Eq. 5 can reveal time periods where P_X is small. The combination of Eq. 9 with thermal inputs made at one or a series of points can also be used [1, 2, 4]. For example, a thermal balance can be made just before application of the heater pulse ($t=t_1$), or near the end of the heater pulse ($t=t_2$), then if the system has relaxed adequately, $dT_{cell}/dt \approx 0$.

Once the accurate determination of $(k'_R)_{262}$ and $C_p M$ is completed for a particular calorimetric cell, then Eq. 9 can be used directly in evaluating the excess power, P_X , during all time periods and even for future experiments using this same calorimetric system. However, the Dewar vacuum may soften with the passage of several years (due mainly to the slow diffusion of

atmospheric helium through the glass walls of the Dewar cell) leading to a gradual increase in the radiative heat transfer coefficient [4].

The application of Eq. 9 to obtain integral heat transfer coefficients requires that the excess power, P_X , be constant over the time period considered. In turn, this requires experiments using suitable “blank” or control systems. If the excess power generation varies with time, one will inevitably conclude that the instrumentation has enhanced errors. Moreover, such a conclusion will apply to any calorimetric system that is used. The lack of execution of appropriate “blank experiments” is undoubtedly a contributory factor to confusion concerning the Fleischmann-Pons calorimetry.

These Dewar calorimeters behave as ideal well-stirred tanks, thus the errors are mainly those set by the temperature measurements. If the temperature measurement error is ± 0.01 K and $T_{\text{cell}} - T_b = 10.00$ K, then this temperature error would be $\pm 0.1\%$. This temperature measurement error can be reduced to ± 0.001 K by calibrating the thermometers, by room temperature control, and by averaging methods; therefore, random measurement errors can be reduced to $\pm 0.01\%$. The use of an 11-point average has been found to be especially useful in decreasing the random errors [4]. For measurements every five minutes, such averaging gives about 52 independent values for the recommended 2-day measurement cycle [4].

The use of a Dewar isoperibolic calorimeter in a control experiment to measure the rate of enthalpy production due to the reduction of electrogenerated oxygen yielded 1.1 ± 0.1 mW where the cell input power was in the range of 800 to 1000 mW [1, 2]. This demonstrates that the calorimetric error was, in fact, reduced to $\pm 0.01\%$ for this experiment [1, 2].

The Use of F-P Dewar Calorimeters at NHE

Three F-P Dewar isoperibolic calorimetric cells with detailed instructions were supplied to the NHE laboratory in Sapporo, Japan around 1993. The NHE studies concluded that the original method of Fleischmann-Pons calorimetry was not satisfactory, hence a modified version of this calorimetry was adopted [8]. It was reported that the NHE modified version of the F-P calorimetry could detect excess power exceeding 30 mW over a two month period whereas NHE claimed an error as large as ± 0.3 W existed for the original F-P method [8].

One of us (M.H.M.) had the opportunity to work directly with this F-P Dewar calorimetry at NHE in 1997-1998 and completed six experiments using these three cells [4, 9-13]. The cathodes investigated were Pd-B, Pd-Ce, and Pd-B-Ce alloys as well as three individual co-deposition experiments [9]. The Pd-B and Pd-B-Ce alloys were prepared by Dr. Imam at the Naval Research Laboratory (NRL), and the Pd-Ce alloy was supplied by Martin Fleischmann. The independent method of data analysis developed by Miles showed that all experiments except the Pd-B-Ce alloy produced significant amounts of excess power and excess enthalpy [9, 10]. The Miles method of analysis used only the experimental data where dT_{cell}/dt was small to minimize the error in the $C_p M$ value (see Eq. 3). This data selection also minimizes the changes of various properties with time and thus minimizes the need for integration of the data to

improve accuracy. By adopting the NHE value of $C_{pM} = 490 \text{ Jmol}^{-1}$ and assuming $P_X = 0$, the lower bound heat transfer coefficient, $(k'_R)_1$, was calculated using Eq. 5. The results for $(k'_R)_1$ for the Pd-B-Ce cell are shown in Fig. 1. Similar calculations of $(k'_R)_1$ were performed for the Pd-B and Pd-Ce cells.

By taking averaged values over selected time periods, the lower bound pseudoradiative heat transfer coefficients were $0.84632 \pm 0.00642 \times 10^{-9} \text{ WK}^{-4}$ for the Pd-Ce-B cell, $0.81120 \pm 0.00976 \times 10^{-9} \text{ WK}^{-4}$ for the Pd-B cell, and $0.79999 \pm 0.00790 \times 10^{-9} \text{ WK}^{-4}$ for the Pd-Ce cell. These cells were labeled A.1, A.2, and A.3, respectively, and placed in the same water bath with cell A.2 in the middle [9]. In retrospect, because these three cells were identical, the larger $(k'_R)_1$ value for the Pd-B-Ce cell, where there was very little excess power, would be more correct. Nevertheless, the individual $(k'_R)_1$ value were used for each cell, and the excess power was then calculated by using Eq. 3. These results are given in Figs. 2, 3, and 4. Large excess power peaks are seen for Pd-Ce (Fig. 2) and Pd-B (Fig. 3), but despite the use of a larger $(k'_R)_1$, there is no obvious excess power for Pd-B-Ce (Fig. 4). All of these calculations were made using Excel spreadsheets.

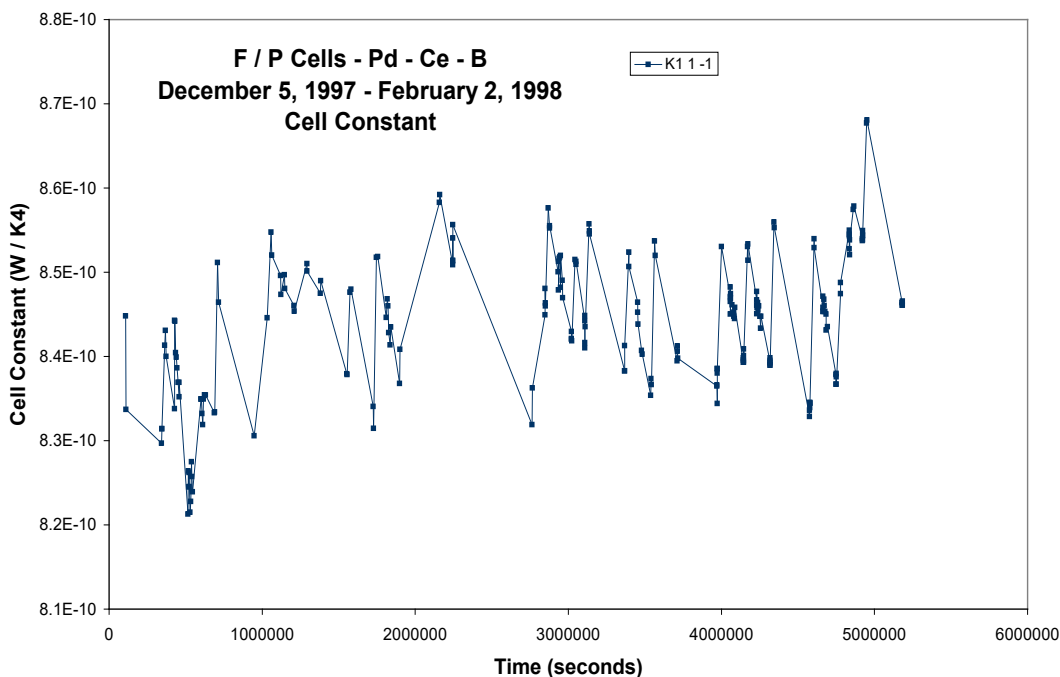


Figure 1. Experimental cell constant, $(k'_R)_1$, versus time for Cell A-1 (Pd-Ce-B).

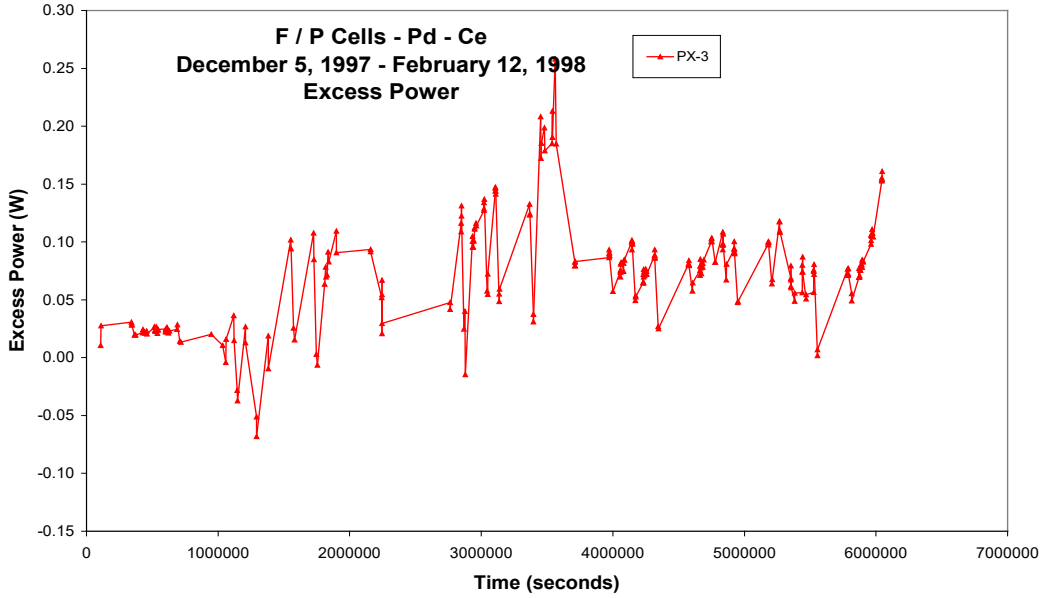


Figure 2. Excess power measurements for the Pd-Ce cathode in Cell A-3.

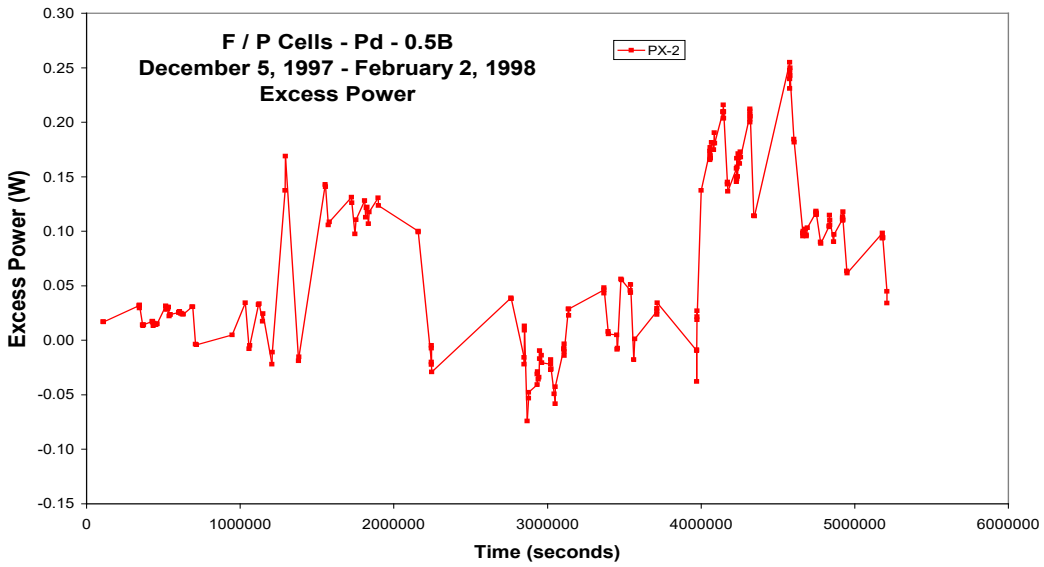


Figure 3. Excess Power measurements for the Pd-B cathode in Cell A-2.

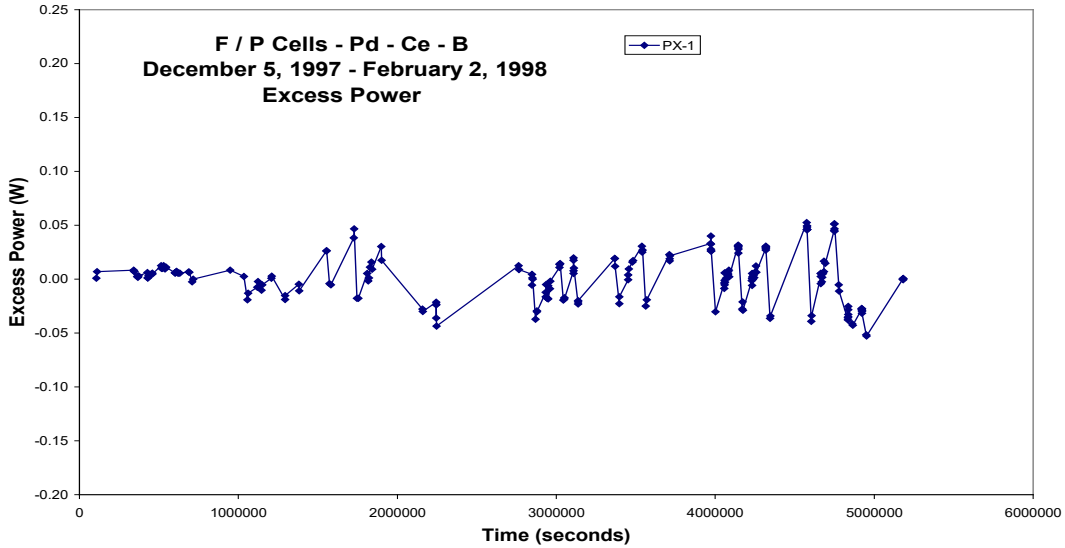


Figure 4. Excess Power measurements for the Pd-Ce-B cathode in Cell A-1.

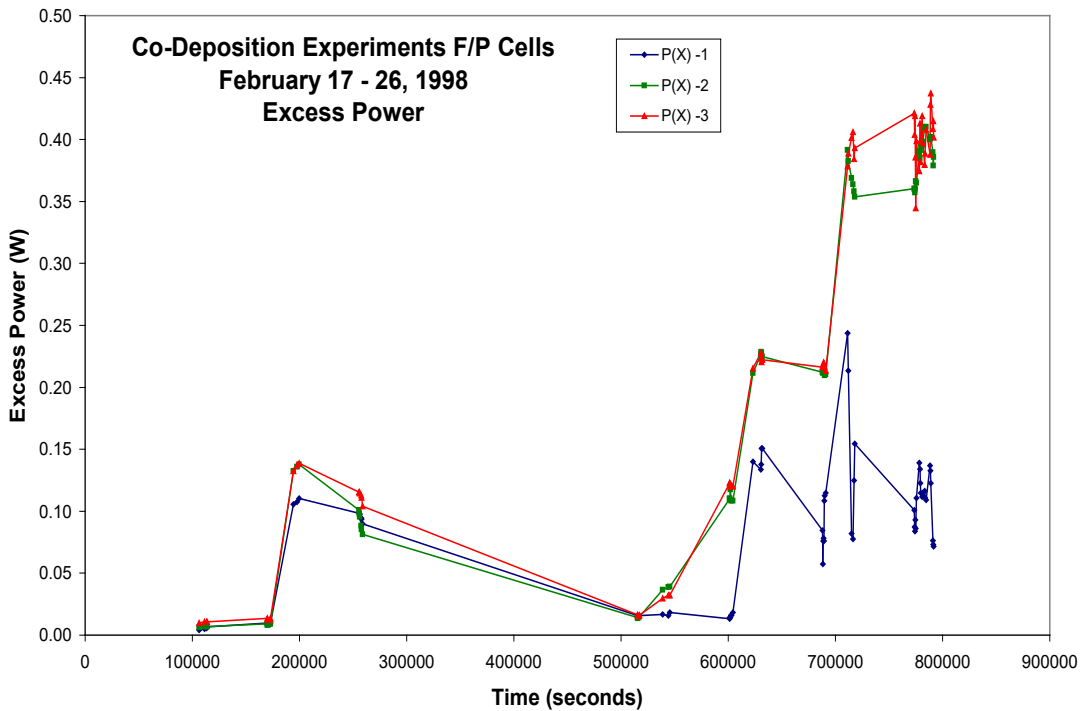


Figure 5. Excess power measurements in three co-deposition experiments.

The following three co-deposition experiments used these same $(k'_R)_1$ values determined previously for each cell along with the adopted C_pM value to calculate the excess power from Eq. 3. As shown in Figure 5, excess power was observed in all three cells and showed similar trends [9-11]. The discontinuity between $t=259200$ s and $t=518700$ s was due to the NHE laboratory closure over the weekend. The only data used in this analysis was the data recorded

by hand in a notebook. The full computer data recorded every five minutes for these six F-P Dewar type experiments became available only after Miles left NHE.

For the three co-deposition experiments shown in Fig. 5, the cell current was turned on to 0.006 A at 86100 seconds, increased to 0.100 A at 173100 seconds, increased to 0.200 A at 259200 s, and then reduced for the weekend due to chlorine evolution. During the following week, the current was increased to 0.100 A at 518700 s, then to 0.200 A at 604500 s, and finally to 0.400 A at 691200 seconds. The larger excess power effects in this F-P Dewar cell were observed at the higher currents. The initial composition of the co-deposition solution was 0.025 M PdCl₂, 0.15 M ND₄Cl, and 0.15 M ND₄OD in D₂O. The cylindrical copper cathodes used in each cell were approximately 4 mm × 25 mm. Careful measurements of changes in electrolyte volume showed that there were no obvious correlations of the excess enthalpy to recombination of the electrogenerated D₂ or O₂ gases [9].

The fact that there was no measurable excess power in the Pd-Ce-B cell (Figure 4) provided an excellent opportunity for the analysis of factors that may affect $(k'_R)_1$ in the F-P type Dewar cells. The factors investigated include the cell temperature, the cell voltage, the volume of electrolyte present in the cell, and the cell current. None of these factors showed a clear relationship with the cell constant. The highest correlation coefficient (R^2) was obtained for the relationship of the cell constant to the cell current, but R^2 is still too small to assign any significance to this trend. Nevertheless, this is shown in Figure 6. It is readily seen that the cell currents were generally 0.400-0.600 A.

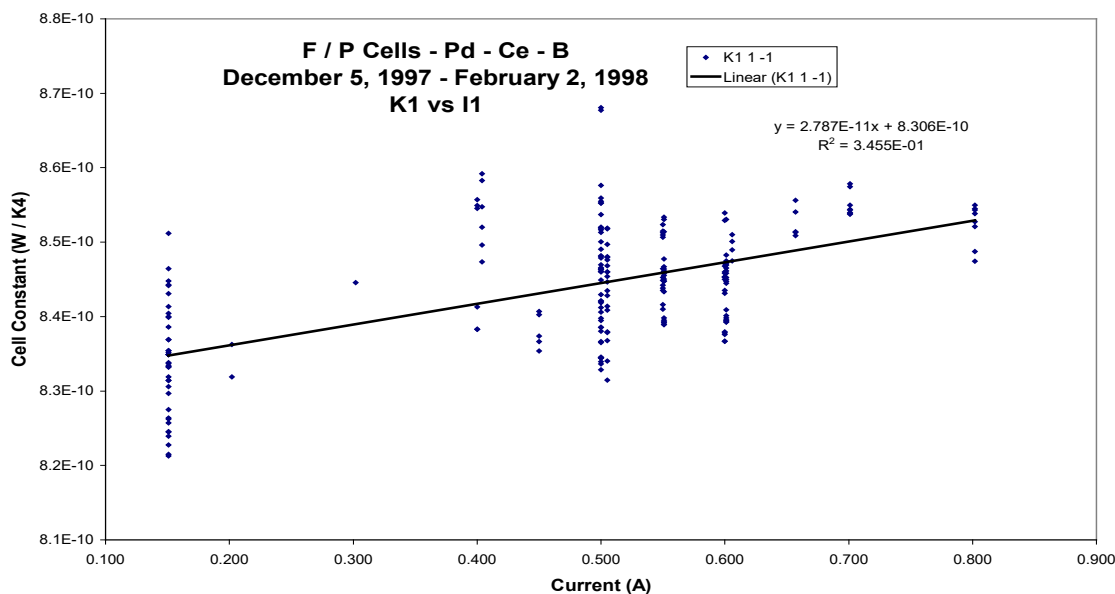


Figure 6. Experimental relationship of $(k'_R)_1$ to the cell current in the Pd-Ce-B experiment.

This cell was initially run for ten days at a current of 0.151 A. The cell constant varied apparently in a random manner from day to day during this time period as shown in Figure 6.

The large variations in the cell constant at this initial low current illustrate the error in using a single heating pulse to determine the cell constant. Experimental data from various time periods is needed to accurately determine the true pseudoradiative heat transfer coefficient [1, 2, 4].

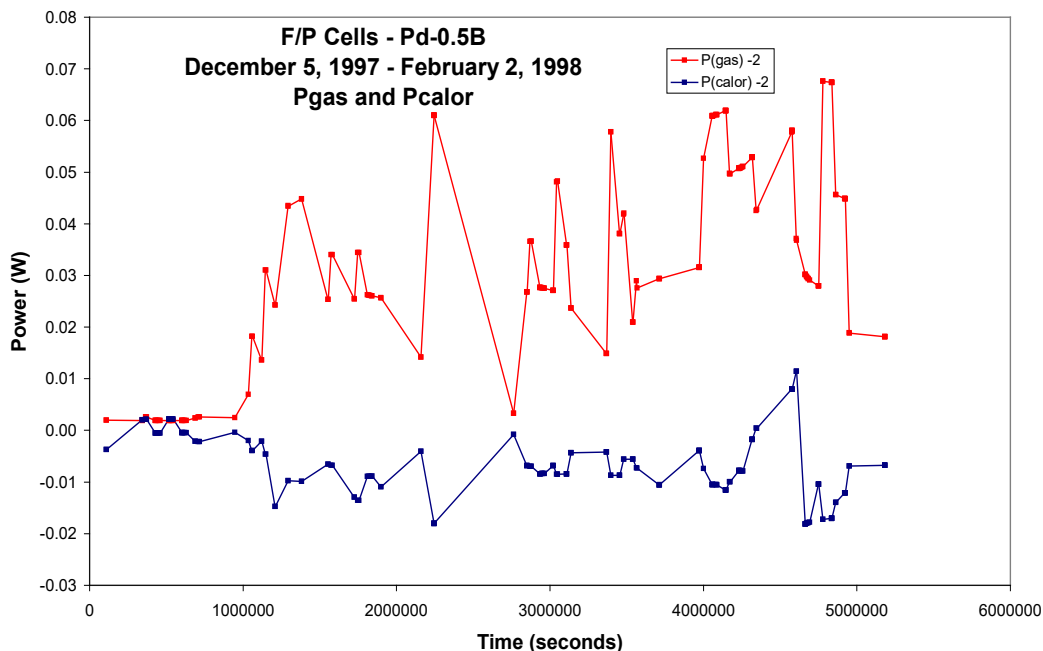


Figure 7. Display of the calorimetric terms P_{gas} and P_{calor} determined from Equations A.2 and A.4 for the Pd-B study.

Figure 7 represents the calculated values for the P_{gas} and P_{calor} terms (Equations A.2, A.4) for the Pd-B study. The P_{calor} term remains small and generally negative under the steady state conditions selected for this data ($dT_{cell}/dt \approx 0$). As seen from Equation A.2, the P_{gas} term will increase with increases in the cell current and cell temperature. Because dT_{cell}/dt was generally less than 10^{-5} Ks^{-1} for the selected data, the error in $C_p M$ (490 versus 450 JK^{-1}) would produce an error in P_{calor} less than 0.4 mW .

A completely independent analysis of the Pd-B cell was later made by Martin Fleischmann using the complete set of the computer data recorded for this experiment. This analysis showed very similar but larger excess power behavior when compared to Fig. 3 for this experiment [4, 7, 12]. This method calculated the true radiative heat transfer coefficient by using the more accurate method of backward integration to yield $(k'_R)_{262} = 0.85065 \times 10^{-9} \text{ WK}^{-4}$ and $C_p M = 450 \text{ JK}^{-1}$ for this cell (see Eq. 9). This k'_R value is in good agreement with the value calculated for the Pd-B-Ce cell where P_X was close to zero (Fig. 1). Furthermore, $C_p M$ is very close to the estimated values based on the mass of the cell components [4]. It is obvious that the method of backward integration of the calorimetric data used by Martin Fleischmann gives the most accurate results.

The NHE method for treating this same experimental data showed only large fluctuations for excess power centered around $P_X = 0$. NHE concluded that the Pd-B, Pd-Ce, Pd-Ce-B and the

co-deposition experiments gave large calorimetric errors and no excess power. A comparison of the NHE and Fleischmann results for the same Pd-B study have been reported previously (see Fig. A.18 and Fig. A.19 of Reference 4).

The main issues with the NHE analysis of the F-P calorimetric data are as follows [4, 7, 12]:

1. The radiative heat transfer coefficient was determined solely by the first heating pulse that occurred on the third day.
2. The presence of any excess heat on the third day will result in radiative heat transfer coefficients that are too small.
3. The use of a radiative heat transfer coefficient that is too small produces, in effect, a shift of the zero line for excess power. Excess power, therefore, becomes confused with the calorimetric error.

The Pd-B study and the three co-deposition experiments were all producing excess power on the third day (see Figs. 3, 5). Another problem was that the heating pulse used by NHE was too short for these cells (6 versus 12 hours); therefore, the cell temperature did not have sufficient time to relax to equilibrium [4, 7, 12]. An additional concern is that forward integration was used by NHE rather than the more accurate backward integration (Eq. 9). Also, the heat capacity of the system (C_pM) used by NHE was too large (490 versus 450 JK⁻¹). This error will become serious for time periods where the cell temperature is changing rapidly with time.

The exact same cell used at NHE should have almost the same radiative heat transfer coefficient. The various values reported are presented in Table I.

Table I. Radiative Heat Transfer Coefficients For The Same F-P Dewar Calorimetric Cell used at NHE in 1997-1998 (Cell A.2).

Method	k'_R (WK ⁻⁴)
Fleischmann (True)	0.85065×10^{-9}
Miles (Lower Bound)	0.8112×10^{-9}
NHE (Pd-B)	0.79350×10^{-9}
NHE (co-deposition)	0.68×10^{-9}
Stefan-Boltzmann	0.754×10^{-9}

If the NHE method were accurate, it would give very close to the same constant for the same cell in each experiment. Instead, there is a 14% difference in the two NHE calculations. Furthermore, the NHE co-deposition value even becomes significantly smaller than the theoretical Stefan-Boltzmann value. This is simply not possible. With a near perfect vacuum in the Dewar and good insulation at the cell top in order to minimize heat losses due to conduction, then the radiative heat transfer coefficient may approach the theoretical Stefan-Boltzmann value from the higher side. Usually it will be a few percent higher due to heat losses via conduction incorporated into k'_R (Eq. 2). With age, the Dewar vacuum will soften due to atmospheric helium diffusion through the glass and into the vacuum, thus k'_R will increase. These F-P cells

were at least 4 years old when used in 1997-1998 at NHE, thus the higher value for k'_R reported by Fleischmann is not surprising. A very similar high value was found for $(k'_R)_1$ from data for the Pd-B-Ce cell ($0.84632 \pm 0.00642 \times 10^{-9} \text{ WK}^{-4}$, see Fig. 1) where there was very little excess power.

The data analysis of these F-P Dewar cells must involve averaging procedures to accurately determine any excess power. For example, an experimental error of only $\pm 0.01^\circ\text{C}$ in a single measurement of the cell temperature produces an error of $\pm 15 \text{ mW}$. This is due to the large value of 450 JK^{-1} for $C_p M$. When this 450 JK^{-1} is multiplied by $\pm 0.01\text{K}/300 \text{ s}$, then $\pm 15 \text{ mW}$ is the error in P_{calor} (Eq. 3). The 300 seconds (5 minutes) is the time period between measurements by the ICARUS data acquisition system.

The Use of F-P Dewar Calorimeters at Grenoble

The objective of the Grenoble (France) work was to check the reliability of the F-P Dewar calorimeter from ambient to the boiling temperature [5]. The Dewar cells with the upper part silvered were claimed to be identical to those used by Fleischmann and Pons [5]. The conclusion of this study over a three year period was that the F-P Dewar calorimeters were very accurate and well adapted to study the F-P effect [5]. Five out of eighteen experiments produced excess heat. The measured excess power using palladium cathodes ranged from 50 to 300 mW. The largest excess heat measurements occurred above 70°C , and the percent of excess during the boiling phase reached 150% [5].

Notable experimental conditions used at Grenoble include the excellent room temperature control ($20^\circ\text{C} \pm 0.1^\circ\text{C}$), the filling of the cell in an argon atmosphere to avoid H_2O and CO_2 contamination, the automatic addition of D_2O every time 1 cm^3 is consumed, the collection of data every 6 seconds, and averaging every minute [5]. The experimental profile was to use a constant current of 0.20 A (0.26 A cm^{-2}) until the cell temperature reached 40°C (typically 1 to 2 weeks). The cell current was then raised to 0.50 A, and the experiment continued until eventually cell boiling began that led to a dry cell [5].

Considerable simplification of the calorimetric equations was used at Grenoble [5]. The excess heat was calculated by neglecting P_H , P_C and P_W in Eq. 1, thus

$$P_{\text{calor}} = P_{EI} + P_X + P_R + P'_{\text{gas}} \quad (10)$$

where P'_{gas} is approximated by using only the larger term that involves the enthalpy of vaporization of D_2O , L (Eq. A.4).

Thus

$$P'_{\text{gas}} = -I/F(0.75P/(P - P^*))L \quad (11)$$

At cell temperatures below 70°C, Eq. 10 is further simplified by assuming that P_{calor} and P_{gas} can also be neglected, thus

$$P_X = -P_R - P_{El} = k_R f(T) - (E - E_H)I \quad (12)$$

The cell was calibrated using a Pt cathode and assuming $P_X=0$, thus Eq. 12 becomes

$$k_R = (E - E_H)I / f(T) \quad (13)$$

Some errors would obviously be introduced by these calorimetric approximations. Nevertheless, these errors would likely be less than 5% and would generally minimize the reported excess power.

A troubling aspect of the Grenoble experiments is that k_R was reported as 0.075, 00905, and 0.078 for cells P1, P2, and P3, respectively, with no assigned units [5]. These values would be more fitting for a conductive heat transfer coefficient, k_C . The electrolyte volumes used in these cells ranged from 72 to 85.5 cm³ giving on average volume of 78.9 cm³ compared to 90 cm³ for F-P Dewar cells used at NHE. If the Grenoble and NHE Dewar cells had a similar diameter, then the radiative heat transfer surface area would be proportional to the cell volumes. Assuming $k_R=0.80 \times 10^{-9}WK^{-4}$ for a newer NHE Dewar cell, then the cells used at Grenoble would have a radiative heat transfer coefficient of $k_R=0.70 \times 10^{-9}WK^{-4}$ instead of the values reported. It is likely that Grenoble converted k_R into an equivalent k_C constant using

$$k_C = k_R f(T) / (T_{cell} - T_b) \quad (14)$$

For a cell temperature of 40°C (313.15 K) and a bath temperature of 20°C (293.15 K), the calculated value for k_C is 0.078 WK⁻¹. This is approximately the numerical values reported by Grenoble [3]. It should be noted that k_C calculated from Eq. 14 will increase with the cell temperature. For $T_{cell} = 70°C$ (343.15 K) and the same bath temperature, $k_C = 0.091$ WK⁻¹. This may explain the range of cell constants reported. Although C_pM was not reported by Grenoble, it can be estimated from the electrolyte volumes that $C_pM = 395$ JK⁻¹ versus $C_pM = 450$ JK⁻¹ for the NHE F-P Dewar cells.

The Use of F-P Dewar Cells at Harwell

The Harwell (U.K.) laboratory investigated the F-P Dewar calorimetric cells during the early stage (1989) of the cold fusion controversy [14]. These Harwell experiments were hastily performed, and the apparent calorimetric error of ±15% falls far short of the ±0.01% error or ±0.1 mW reported by Fleischmann [1, 2].

The F-P type Dewar calorimeter used by Harwell had an inner diameter of 2.5 cm and a height of 10.0 cm with no silvering versus 2.5 cm and 25.0 cm respectively, with silvering for the ICARUS-1 cell used at NHE. From the cell dimensions given, it appears that stirring by the electrolysis gases would be adequate for the Harwell cell. A single thermistor was used that was

positioned in the upper half of the cell. Details of this Harwell cell design have been presented elsewhere [14, 15].

The Harwell publication [14] provides only fragmental information about the calorimetric equations used in their calculations for the F-P Dewar cell. Searching for terms corresponding to Eq. 1 yielded only P_{EI} and an approximation for P_{gas} that is even less accurate than Eq. 11. The P_{calor} , P_H , and P_W terms are missing. The missing P_{calor} term may account for periods of large endothermic behavior (Figs. 2b, 2c, 2d of Ref. 12) that violates the First Law of Thermodynamics. About equal contributions were reported for P_R and P_C modeled simply by

$$P_R + P_C = k \Delta T \quad (15)$$

where $k = 0.1 \text{ WK}^{-1}$ [14]. If true, this suggests a very poor vacuum for the Dewar cell that was used at Harwell.

To the credit of the Harwell group, their raw data was made available, and an independent analysis of this raw data has been reported by Melich and Hansen [16, 17]. This analysis reduced the calorimetric error to approximately 1% for some of the time periods. In one of Harwell's D_2O cells (Cell 3), there were more than ten time intervals where an unexplained power source may be operating in the F-P cell to produce 100 to 200 mW of anomalous power [16]. It was also concluded that Harwell would not have been expected to produce significant excess heat in almost all of their cells because of low current densities, low levels of D loading into the Pd, and other poor choices in their experimental design [15].

Another important analysis of the Harwell raw data has been made by Fleischmann [15]. Seventeen "heat bursts" were documented for a Pd/ D_2O +0.1 M NaOD study (Cell 3) whereas none were observed for a similar Pd/ H_2O + 0.1 M NaOH experiment (Cell 4). The calculation of $(k'_R)_1$, by assuming $P_X = 0$ (Eq. 5) for the Harwell data shows a significant difference in actual values as well as different behavior over time for the H_2O and D_2O experiments [15]. Because P_X is actually close to zero for the H_2O study, then $(k'_R)_1 = (k'_R)_2$ for this experiment in the Dewar cell. The use of Eq. 7, therefore, gives an excess power of $P_X = 190 \text{ mW}$ at 14239 hours that decreases to $P_X = 70 \text{ mW}$ at 15636 hours for the D_2O study (Fig. 3A, Ref. 15). Fleischmann also concluded that this D_2O study shows a decrease in excess power with time [15]. Fleischmann's final conclusion was that excess enthalpy generation was in fact observed in the Harwell study contrary to the conclusions reached by the authors [15].

Discussion of possible errors in the Harwell experiments has also been provided by Worledge [18]. Many of the Harwell experiments were run at current densities below the threshold required for excess power production [18]. The persistence of Harwell's negative power balance is not easily explained [18]. To stabilize the baseline during calibration of the F-P cell, the electrolytic current used by Harwell was reduced to zero or to 20-40% of the operating current [18]. Uncontrolled errors could be introduced by changing the cell current to perform these calibrations [18]. It was also shown that the assertion in the Harwell paper that use of the linearized form (Eq. 15) always overestimates the cell power is incorrect [18].

The Harwell laboratory used two other calorimeters in addition to the F-P Dewar cell. These were the improved heat-flow calorimeter (IHF) with a 500 mL capacity and an isothermal calorimeter with a 1000 mL capacity [14]. The later calorimeter was designed to determine the heat production for samples of plutonium, thus the operating environment for the F-P experiment using this system was quite different from that for Dewar cells. As admitted by Harwell [14], the large volumes of these cells makes them less sensitive for the detection of any excess power.

A previous summary of error sources and problems for the Harwell calorimetry included their method of cell calibration during electrolysis when any excess power is unknown, their large power changes during calibrations, marked endothermic behavior following topping their cells with D₂O, and their use of small electrodes in large electrolyte volumes that would minimize the detectability of any excess power effect as well as contribute to poor stirring [19]. Furthermore, the unfavorable geometry of various cathodes (beads, ribbon, bar) would not provide for uniform electric fields and symmetry required for high deuterium loadings [19]. As previously concluded, the Harwell results in no way disprove the F-P effect [17].

China Lake Calorimetry / Heat Conduction

No Dewar cell was used in the China Lake isoperibolic calorimeter, thus the main heat transfer pathway was by conduction through the insulated jacket surrounding the cell. The same calorimetric equations apply except Eq. 2 becomes

$$P'_C = P_C + P_R + P_W = -k'_C(T_{cell} - T_b) \quad (16)$$

where the much smaller P_R and P_W terms are incorporated into a pseudoconductive power term, P'_C . Therefore, Eq. 3 becomes

$$P_{calor} = C_p M (dT_{cell} / dt) = P_{El} + P_H + P_X + P_{gas} - k'_C \Delta T \quad (17)$$

where $\Delta T = T_{cell} - T_b$.

The basic design of the China Lake calorimeter has been described in previous publications [20-22]. Important features include the small size of the electrochemical cell (1.8 cm diameter and 15.0 cm length) that was filled with 18.0 cm³ of electrolyte. The electrochemical cell was placed in a calorimetric jacket within a secondary compartment that contains two thermistor positioned outside the cell on opposite sides of the cell wall and at different heights from the bottom of the cell (1.9 and 4.5 cm). This calorimetric design where the temperature is measured in a secondary liquid or solid phase surrounding the electrochemical cell minimizes the sloping baseline problem, i.e. the change in the cell constant due to solvent losses [19]. The cell can then be simply modeled as the heating source for the secondary phase. This secondary phase also serves as an integrator for the power produced by the electrochemical cell. The estimated error range of the basic China Lake calorimetry was ± 20 mW or $\pm 1\%$ of the input power, whichever is larger [19, 22]. The pseudoconductive heat transfer coefficient varied somewhat with the actual cell used, but generally k'_C was approximately 0.14 WK⁻¹[19]. The value for $C_p M$ was estimated at 150 JK⁻¹. Calorimetric measurements were generally performed where dT_{cell}/dt was small, thus

P_{calor} was generally less than 10 mW and usually negative in sign [19]. No mathematical integration of the differential equation (Eq. 17) was used to improve the accuracy.

Near the end of the Navy (ONR) funded program at China Lake, an improved calorimeter was developed with the work of Kendall B. Johnson [20, 23]. The noise level for this improved calorimeter was only 0.2 mW or 0.1% of the input power, whichever is larger. This new open, isoperibolic calorimeter used at China Lake along with the specific improvements is illustrated in Figure 8.

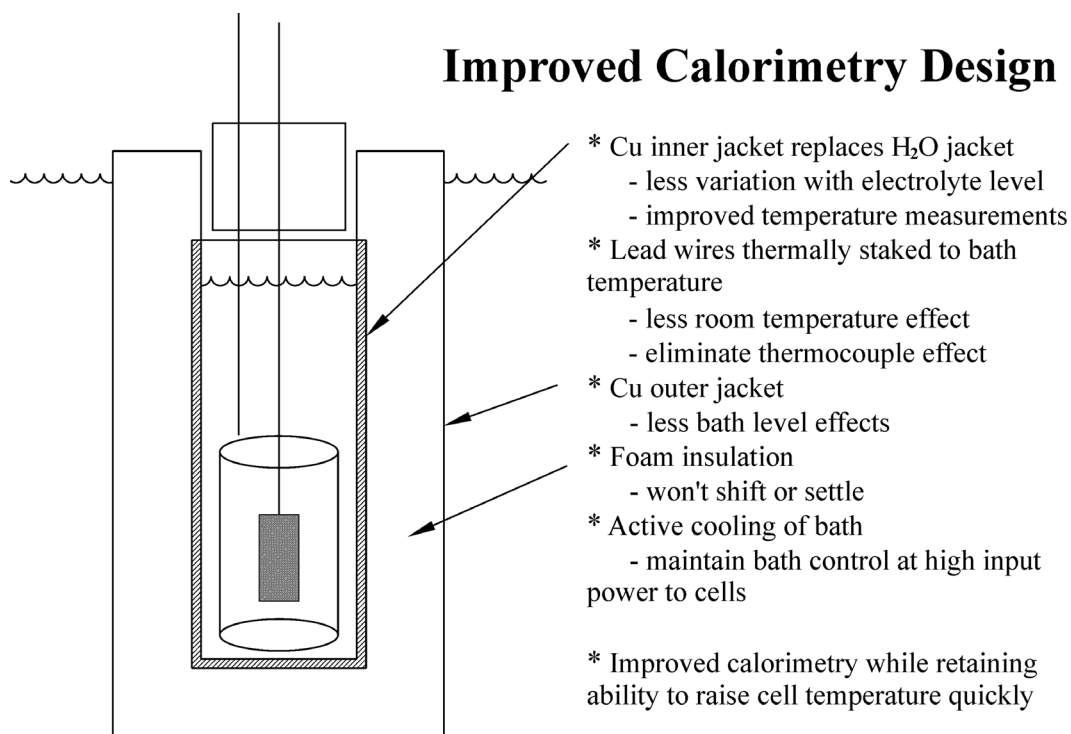


Figure 8. Improved design for the integrating, open, isoperibolic calorimeter constructed at China Lake in 1994.

The major new improvements included a copper inner jacket that acts as the integrator and replaces the previous H₂O-filled jacket. An insulating box over the cell top and bath greatly reduced the effect of changes in the room temperature. A copper outer jacket contacts the bath and minimizes bath level effects by virtue of its high thermal conductivity. Foam insulation was used between the two copper jackets to avoid any shifting of the insulating material. Furthermore, the lead wires coming out of the cell were thermally staked to the bath itself, thereby further reducing the effect of the room temperature. Active cooling of the bath provided excellent stability for the bath temperature even at high input power to the cell. The size of the electrochemical cell remained the same and provided a filled volume of 18 cm³ for the electrolyte.

The ability of this new China Lake isoperibolic calorimeter to measure the heat of absorption of deuterium into the palladium cathode is shown in Figure 9.

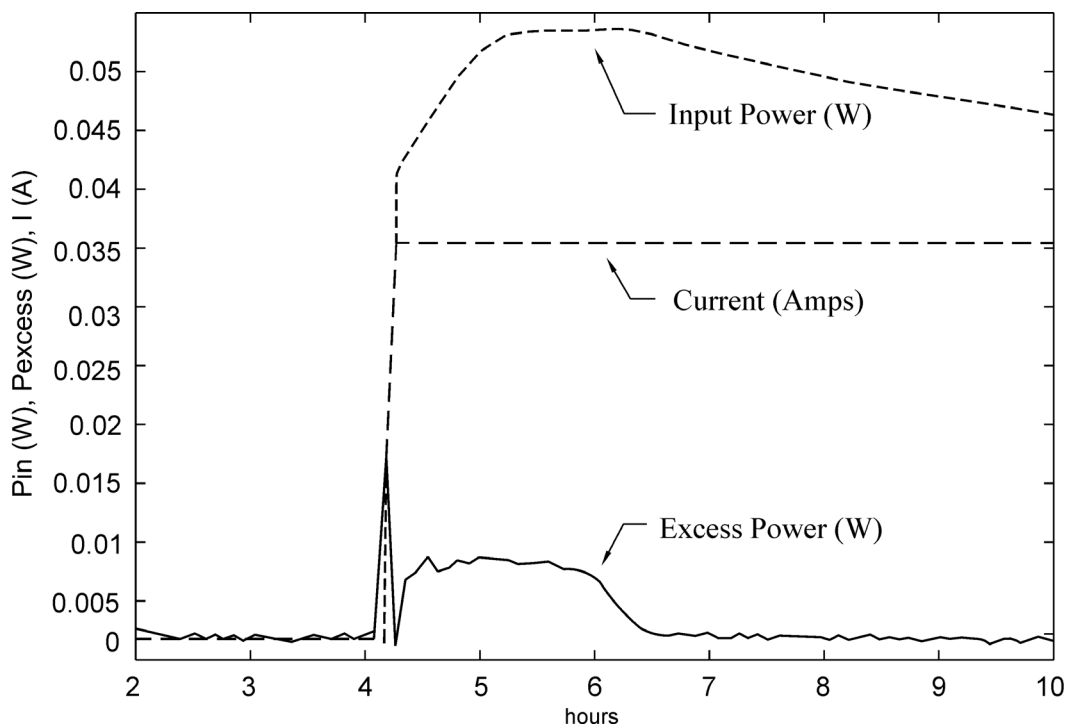


Figure 9. Measurement of the exothermic heat of absorption of deuterium into a palladium wire (0.1×4.3 cm) cathode using the improved China Lake calorimeter.

An excess power output of approximately 6.5 mW was observed for 2 hours (Figure 9). Integrating the excess power shown in Figure 9 yields -44.7 J that compares very favorably to the calculated -40.2 J based on the cathode size (0.1×4.3 cm Pd wire), a loading level of PdD_{0.6}, and using the reported value of $\Delta H = -10.53$ kJ/mol Pd [20]. The somewhat larger experimental value is expected because the loading shown in Figure 9 likely proceeds beyond PdD_{0.6} to generate even more heat. Assuming ΔH remains constant, the measured excess power would correspond to PdD_{0.67}.

Several excess power effects were measured with this improved China Lake calorimeter before the ONR funded program terminated in 1995 [20]. Figure 10 presents an excess power effect of approximately 75 mW for a Johnson-Matthey wire cathode sample. Previous samples of this material had produced excess heat in earlier China Lake experiments with the older calorimetric design [20]. A later experiment at NHE in 1997 using this material also produced excess power [21].

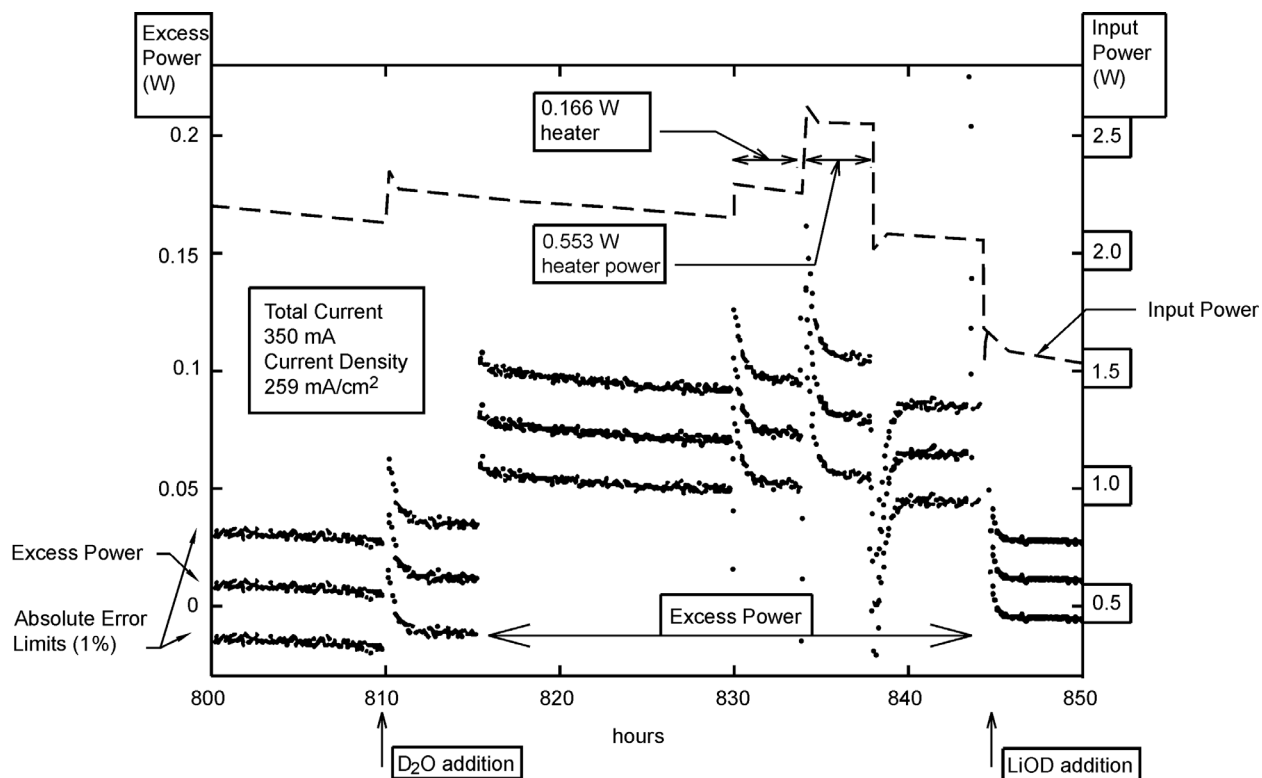


Figure 10. Excess power measurements for a Johnson-Matthey wire cathode (0.1×4.3 cm) as measured with the improved China Lake calorimeter.

The onset of the excess power effect in Figure 10 occurs rather suddenly at about 815 hours. The loss of this F-P effect at 844 hours was also sudden, but this effect correlated to the addition of LiOD that caused a sharp decrease in the input power (Figure 10). A major advantage of this calorimetry was its small time constant that allows prompt measurements of changes in the excess power. Unfortunately, no further experiments with this improved China Lake calorimetry were possible despite the extensive work by Kendall Johnson that went into the designing and building of this calorimeter with its associated data acquisition system. With the loss of the ONR funding, management at China Lake dictated that no further work on the F-P effect was to be done. Dr. Johnson moved on to a position in Idaho, and Dr. Miles was assigned by the Head of the Chemistry Department at China Lake (Dr. Robin A. Nissan) to report to the stockroom clerk for the inventory of chemicals [24]. No further studies of the F-P effect were made at China Lake after 1995. Even the writing and publishing of the China Lake final report [20] was not possible until outside funding was provided in 1996 by Dr. Dave Nagel of NRL.

Recent anonymous funding has provided for the construction of four similar improved calorimeters with a larger cell size ($2.5 \text{ cm} \times 20 \text{ cm}$). Experiments are in progress using these new calorimeters.

California Institute of Technology Calorimetry

The initial publication by Caltech on the F-P effect was received by Nature on 23 May 1989 [25]. This means that all work was completed in less than two months after the initial announcement of the F-P effect (March 23, 1989). It is remarkable that Caltech claims completion of multiple calorimetric experiments in this short time span because completion of a single F-P experiment generally requires 4 to 8 weeks of electrolysis. It was reported that no evidence had been obtained for any excess enthalpy, neutron, gamma ray, tritium or helium production during electrolysis of D₂O with palladium cathodes [25].

The first step in evaluating this Caltech calorimetry is to look for the modeling equations such as Eq. 3 or Eq. 17. It was found that this paper is almost devoid of any calorimetric equations. Only P_{EI} and a term for the total power (P_T) could be identified where $P_T = (T_{cell} - T_b) / h$, thus $h = K_C^{-1}$ (KW⁻¹) and is called the heating coefficient. This use of h shows that the main heat transfer pathway was by conduction in the Caltech calorimetry. A Dewar flask containing 30 mL of electrolyte was used, but the Dewar walls contained 1 atm of air, hence there was no vacuum and heat conduction would dominate over electromagnetic radiation.

A Tronac Model 1250 vacuum-jacketed calorimetric Dewar is also described, but it appears that most reported results were for the air-jacketed Dewar [25]. A surprising feature was that the electrolyte was vigorously stirred with a motor, and the resulting Joule heating from stirring (~0.3°C) had to be accounted for in all measurements [25]. Besides being a rather large error source, this need for stirring suggests a much larger diameter cell than the NHE, Grenoble, or China Lake cells. A schematic of the calorimeter in a later Caltech publication [26] indeed shows a short, fat cell with a length/diameter ratio of only 1.4 versus a ratio of 10 (25 cm / 2.5 cm) for the NHE F-P cells [4]. There would not be room for the unneeded mechanical stirrer in the narrow F-P Dewar cells.

The most disturbing aspect of the Caltech report concerns the heating coefficient in a Pd/D₂O+0.1 M LiOD experiment that was allowed to increase with time from 14.0 KW⁻¹ (0.0714 WK⁻¹) at 14.7 hours to 15.9 (0.0629 WK⁻¹) KW⁻¹ at 115.0 hours. This could just as well have been interpreted as a 13.6% excess power effect. Experiments at Caltech for Pd/H₂O + 0.1 M LiOH gave lower heating coefficients ($h = 12.5 \pm 0.7$ KW⁻¹) that did not show this large increase with time. Using a constant h value of 14.0 KW⁻¹ yields excess power that increases with the electrolysis time up to 76 mW [19]. The excess power density of 1.0 W/cm³ Pd for an analysis of the Lewis study [19] is in excellent agreement with reported excess power in other F-P experiments using similar current densities [27, 28]. A complete analysis of the Caltech calorimetry has been previously presented [19]. Caltech was informed of this error analysis and was asked to respond, but no rebuttal was received. A related discussion of the Caltech calorimetry was also produced by Noninski [29]. Both Noninski [29] and Miles [19] concluded that the Caltech calorimetric method of replacing a portion of the electrolysis power by resistor power cannot be used to prove that there is no excess power. This Caltech method only indicates that any resulting change in excess power is small.

Another related paper by Caltech that appeared later in 1989 [26] provides no additional information on the calorimetric equations used to model the calorimeter except for

$$P_{EI} = (E - \gamma E_H) I \quad (18)$$

where γ is the current efficiency for electrolysis. If all the electrolysis gases escape from the open cell, then $\gamma = 1.00$. This is the case in almost all F-P experiments in open cells, thus Eq. A.3 can be used. Nevertheless, the current efficiency should always be monitored to justify the use of $\gamma = 1.00$ (Eq. A.3). In several co-deposition experiments at China Lake where palladium particles became exposed to the electrolysis gases above the electrolyte level, γ values considerably less than unity were measured and appropriate corrections were made [20]. For a control experiment using Pt/D₂O+0.1 M LiOD in a F-P Dewar cell, the power due to the reduction of electrogenerated oxygen at the cathode was measured at 0.0011 ± 0.0001 W with $I=0.2000$ A [1, 2]. Therefore, γ was very close to unity for this cell.

Massachusetts Institute of Technology Calorimetry

The stated purpose of the M.I.T. investigation was to replicate as nearly as possible the experimental procedure of Fleischmann, Pons, and Hawkins [30]. Their conclusions stated that no excess output power or any other evidence of fusion products (neutrons, gamma, helium, tritium) was detected [30].

For both the Phase I and Phase II calorimetry, Dewar cells were not used, thus the main heat transfer pathway was by conduction rather than by electromagnetic radiation as in the F-P Dewar cells. The Phase I calorimetry used by M.I.T. was admittedly relatively crude with error limits of ± 0.3 W. The two Phase II calorimeters were greatly improved and consisted of reasonable cell dimensions of 12 cm in height and a narrow 2.1 cm in diameter. For these two cells, the electrolysis gases provided sufficient mixing of the electrolyte to provide thermal uniformity for cell current densities above about 18 mA/cm² (50 mA). Unlike the short, fat cells used by Caltech [25, 26], no mechanical cell stirring was necessary.

The two M.I.T. calorimeters used glass wool thermal insulation in the compartment between the cell and water bath [30]. The Pt RTD thermometer used in each cell by M.I.T. had an accuracy of $\pm 0.1^\circ\text{C}$ compared to $\pm 0.01^\circ\text{C}$ for the two thermistors used in each F-P Dewar cell. The sensitivity of the Phase II M.I.T. calorimeters was stated at ± 40 mW [30] contrasted to ± 0.1 mW sensitivity for the F-P Dewar calorimeters [1, 2]. From the dimensions of the M.I.T. Pd rod cathodes (0.1×9 cm), the expected excess power of about 70 mW would not be easily distinguished from the ± 40 mW calorimetric error. The M.I.T. laboratory reported their key calorimetric measurements over a rather short time period (100 hours). The observation of measureable excess power with Pd cathodes in D₂O generally requires 6 days or more of electrolysis [19]. No data was provided by M.I.T. for the bath temperature used or for their cell constants (k_C), hence it is not possible to accurately check their calorimetric results.

The proper calorimetric equation to be used by M.I.T. would be Eq. 17. However, M.I.T. used a feedback control system that adjusted the heater power to maintain a constant cell temperature

($dT_{cell}/dt = 0$), thus $P_{calor} = 0$ (Eq. 17). The calorimetric equation reported by M.I.T. [30] transforms into

$$P_{calor} = 0 = P_{EI} + P_H + P_X - k_C \Delta T \quad (19)$$

Comparison with Eq. 17 reveals that the P_{gas} term (Eq. A.4) involving mainly the evaporation of D₂O is missing. Nevertheless, this error would be constant for a constant cell temperature and cell current (Eq. A.4). Although no equation for P_{gas} was presented, M.I.T. comments that evaporative losses will affect power measurements for the calorimetric cell [30]. It was assumed by M.I.T. that at constant cell temperature

$$P_X + P_H = k_C \Delta T - P_{EI} = constant \quad (20)$$

thus a decrease in P_H at constant T_{cell} would measure any increase in P_X . This is only an approximation because the cell voltage, E , and hence P_{EI} (Eq. A.3) will decrease with time due to the decreasing electrolyte volume and hence increasing conductance. Furthermore, the decreasing electrolyte volume will cause a decreasing cell constant, k_C [19]. Therefore, it is not unexpected that a significant drift in P_H caused by the reduction in solvent volume was observed over long time periods [30]. This drift in P_H over time makes the determination of excess power using Eq. 20 difficult and subjective.

It is interesting that the M.I.T. D₂O cell (Fig. 5 of Ref. 30) shows an increasing cell temperature and a corresponding decreasing cell voltage that was not present in the H₂O cell (Fig. 4 of Ref. 30). This suggests a possible excess power effect in the D₂O cell that increases with time. The feedback system should have lowered the heater power (P_H) to maintain a constant temperature in the D₂O cell, but (heaven forbid) this would have implied an increase in excess power using Eq. 20. This increase in cell temperature of 0.3°C gives an estimated excess power of 30 mW (assuming $k_C = 0.1$ W/K) that was not recorded by Equation 20. It has been claimed that there was a serious discrepancy between the unpublished raw data and the final published data for the M.I.T. D₂O cell [31]. The D₂O data points appear to be arbitrarily shifted down to make the apparent excess power vanish [31]. Extensive analysis performed by Dr. Mitchell R. Swartz concludes that the M.I.T. H₂O and D₂O data sets were “asymmetrically” treated [32-34]. This controversy caused M.I.T. to change their calorimetric conclusion from the previous “failure-to-reproduce” to “too-insensitive-to-confirm” [33].

A simple method to ascertain that the H₂O and D₂O data sets were not treated equally by M.I.T. has been pointed out by Jed Rothwell [35]. It was reported that both the H₂O and D₂O raw data was averaged over 1-hour blocks to produce the two figures [30]. For the H₂O data (Fig. 4 of Ref. 30), it is readily verified that the averaged data points are evenly spaced with one point per hour as stated. Even a cursory visual inspection shows that this is not true for the D₂O data (Fig. 5 of Ref. 30). The unpublished M.I.T. raw experimental data as recorded for the D₂O cell is presented in Figure 11. Even if one ignores the raw data showing 235 mW of average excess power over the first 13.6 hours that was adjusted to zero in Figure 11, there remains later peaks that yield about 70 mW of excess power. The published data for this D₂O cell is shown in Figure 12 where the averaged data points for the 1-hour blocks were apparently moved horizontally as well as vertically to give the unequal spacing and near zero excess power [35].

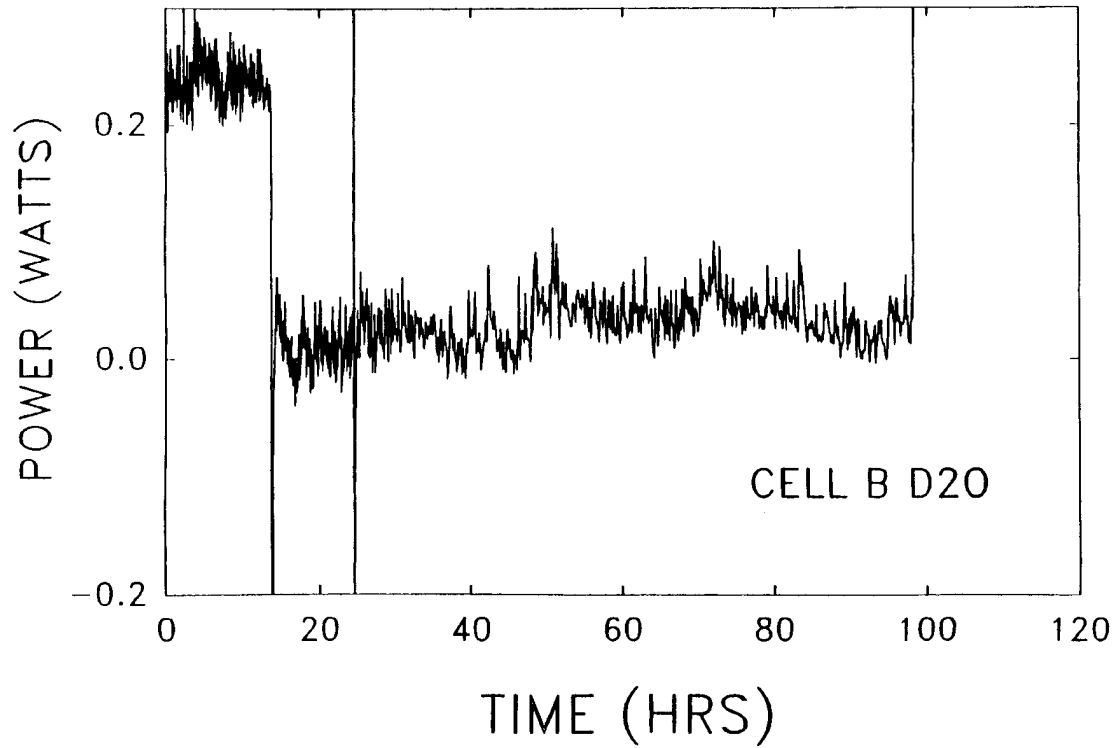


Figure 11. Raw Experiment data as recorded for the M.I.T. D₂O cell.

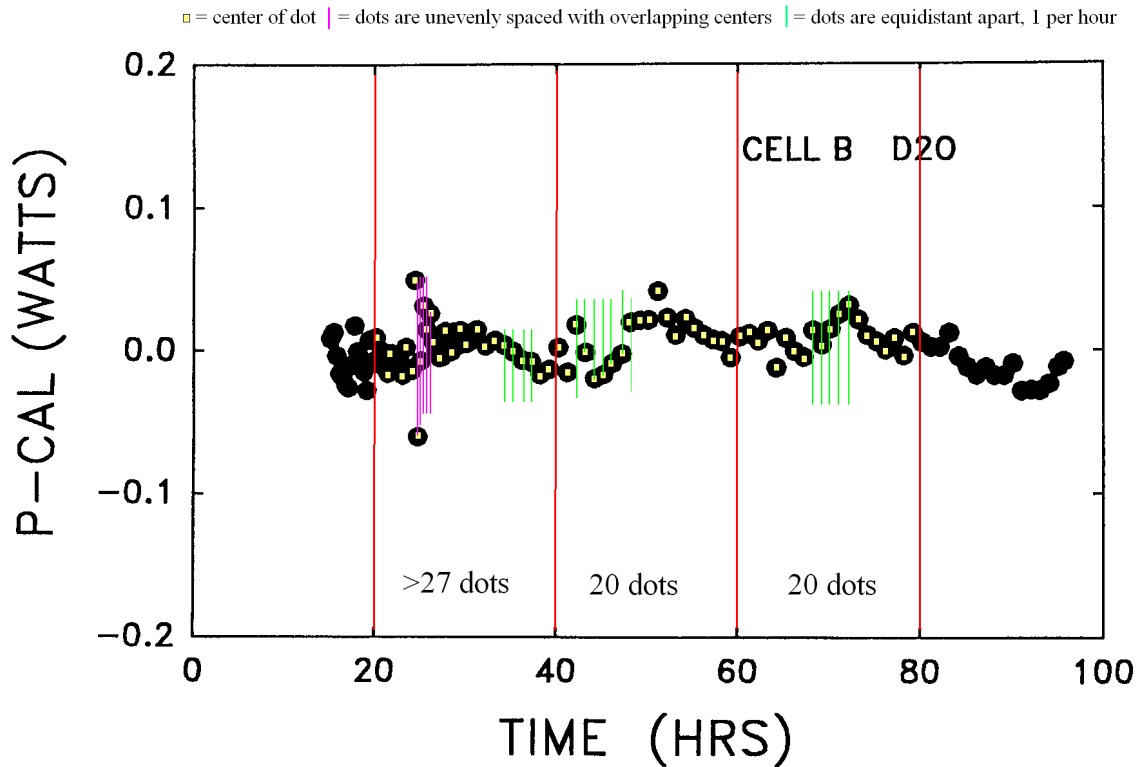


Figure 12. The M.I. T. D₂O data as published; averaged over 1-hour blocks. Note the unequal horizontal time spacing of the data points (Figure provided by Jed Rothwell).

It is difficult to explain the D₂O data points in Figure 12 relative to the raw data in Figure 11 other than the manipulation by M.I.T. of these results. The announced “failure to confirm” by the M.I.T. group became one of the three top negative reports, along with Harwell and Caltech, that strongly influenced the perception of cold fusion in 1989. The M.I.T. group’s report was the first technical reference cited in the influential DOE Cold Fusion Panel’s report in November, 1989 [36].

Summary

The correct equations for modeling isoperibolic calorimetry using open cells are now well established and can give highly accurate results. There has been no challenge to these calorimetric equations in any refereed scientific publication even after nineteen years. Other advantages of this isoperibolic calorimetry for studies of the Fleischmann-Pons Effect include a wide dynamic range, direct visual observations inside the cell during calorimetric experiments, low cost, self-purification (removed of H), and safe operation. These calorimetric equations were used to evaluate the Harwell, Caltech, and M.I.T. calorimetry performed in 1989. It appears that objectivity was sacrificed by these three influential institutions in order to obtain the desired result of no anomalous power effects. The calorimetry used at each of these institutions was notably inferior to that used by Fleischmann and Pons.

Acknowledgements

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APPENDIX I. Calorimetric Equations

Treating the isoperibolic calorimetric cell as the system of interest, the First Law of Thermodynamics expressed as power (J/s or W) becomes

$$P_{calor}(t) = P_{EI}(t) + P_H(t) + P_X(t) + P_{gas}(t) + P_R(t) + P_C(t) + P_W(t) \quad (A.1)$$

By definition, P_{calor} is the power for the calorimetric cell, P_{EI} is the electrochemical power, P_H is the internal heater power, P_X is any anomalous excess power, P_{gas} is the power carried away by the gas stream exiting the open cell (D₂, O₂, D₂O vapor), P_R is the net power transferred by radiation between the cell and water bath, P_C is the power transferred by conduction, and P_W represents the rate of pressure-volume work by the generated electrolysis gases (D₂, O₂). Because both the cell temperature and cell voltage change with time during electrolysis, most of the terms in Eq. A.1 also vary with the time (t). The internal heater power, P_H , will be zero except for the time period between turning the heater on (t_1) and off (t_2). Typical values for P_H , when on, are either 0.2000 W or 0.2500 W. There are often time periods where P_X is zero or constant. As usual in thermodynamics, positive quantities represent power added to the system (calorimetric cell) and negative quantities represent power given off by the system to the surroundings.

The mathematical expressions for the terms in Eq. A.1 are as follows:

$$P_{calor} = C_p M (dT_{cell}/dt) \quad (A.2)$$

$$P_{EI} = (E - E_H) I \quad (A.3)$$

$$P_{gas} = -(I/F) \{ [0.5 C_{P,D_2} + 0.25 C_{P,O_2} + 0.75 (\mathbf{P}/(\mathbf{P}^* - \mathbf{P})) C_{P,D_2O(g)}] \Delta T + 0.75 (\mathbf{P} / (\mathbf{P}^* - \mathbf{P})) L \} \quad \text{where } \Delta T = T_{cell} - T_b \quad (A.4)$$

$$P_R = -k_R f(T) \quad \text{where } f(T) = T_{cell}^4 - T_b^4 \quad (A.5)$$

$$P_C = -k_C (T_{cell} - T_b) \quad (A.6)$$

$$P_W = -RT_{cell} (dn_g/dt) = -RT_{cell} (0.75 I/F) \quad (A.7)$$

The pressure terms in Eq. A.4 are bolded to minimize confusion with any power terms. Under normal constant current (I) operation, the terms T_{cell} , E , ΔT , and \mathbf{P} will vary with time, t . The change of the cell temperature with time, as given by P_{calor} in Eq. A.2, makes Eq. A.1 a nonlinear, inhomogeneous differential equation. Although this differential equation can be used directly, numerical integration yields more accurate results. The P_{calor} term is obviously larger when the cell temperature is changing more rapidly such as when D₂O is replenished or when the internal heater is turned on (t_1) or off (t_2). The normal 2-day experimental cycle for isoperibolic Dewar cells is to replenish D₂O at $t=0$, turn on the internal heater at $t_1=12$ hours, and turn off the

internal heater at $t_2=24$ hours for the next 24 hours. This sequence allows adequate time for the relaxation of the cell temperature following each change back to the small baseline value for dT_{cell}/dt . The small progressive decrease with time for the cell temperature is due to the progressive increase of the electrolyte concentration due to electrolysis resulting in an increasing ionic conductance and hence a decreasing cell voltage and input power (Eq. A.3).

The most complicated term, P_{gas} , is generally small except when the cell temperature exceeds 70°C. The electrolysis reaction



consumes 0.5 mol of D₂O and produces 0.5 mol of D₂ gas and 0.25 mol of O₂ gas per Faraday, F (96485.3415 C mol⁻¹). The 0.75 moles total of electrolysis gases generated per Faraday also carry away D₂O vapor due to the equilibrium vapor pressure of D₂O in the cell, $P = P_{D_2O_{(g)}}$. Using Dalton's Law of partial pressures, the moles of D₂O_(g) carried away per Faraday are given by

$$\text{moles } D_2O_{(g)} = 0.75 (P/(P_{D_2} + P_{O_2})) = 0.75 (P/(P^* - P)) \quad (A.9)$$

where the gas pressure within the cell, P^* , is expressed by

$$P^* = P_{D_{2(g)}} + P_{O_{2(g)}} + P_{D_2O_{(g)}} \quad (A.10)$$

and is close to the atmospheric pressure for this open system. This P^* term dictates the monitoring of the atmospheric pressure for highly accurate calorimetric measurements, but this has not been done by most laboratories. The largest term for P_{gas} results from the large enthalpy of vaporization of D₂O, $L=41678.9$ J/mol at 101.42°C. Therefore, simpler versions of Eq. A.4 are often used such as

$$P_{gas} = -(0.75 I/F)(P/(P^*-P))[(C_{p,D_2O_{(g)}} - C_{p,D_2O_{(l)}})\Delta T + L] \quad (A.11)$$

Assuming $I=0.2000A$ and $T_b=22.00^\circ C$, the P_{gas} term is -5.6489 mW at $T_{cell}=40.00^\circ C$ and increases to -57.8253 mW at $T_{cell}= 80.00^\circ C$. The term involving the enthalpy of vaporization (L) contributes -4.9103 mW and -52.9857 mW, respectively, at these two cell temperatures, (Ref. 2). The electrolysis of D₂O in the calorimeter causes the equivalent mass to change with time

$$M = M^\circ - (1+\beta) \gamma It/2F \quad (A.12)$$

where I is the constant current, β is the fraction of D₂O lost by evaporation, and γ is the current efficiency for electrolysis. Thus, a more exact expression used in earlier publication by Fleischmann and Pons is

$$P_{calor} = C_p d/dt (M\Delta T) = C_p M(d\Delta T/dt) + C_p \Delta T(dM/dt) \quad (A.13)$$

or equivalently

$$P_{calor} = C_p [M^\circ - (l+\beta) \gamma It/2F](d\Delta T/dt) - C_p \Delta T (l+\beta) \gamma I/2F \quad (\text{A.14})$$

where $\Delta T = T_{cell} - T_b$. For constant T_b , $d\Delta T/dt = dT_{cell}/dt$. Except for small cells and large currents, the simpler Eq. A.2 is adequate.

In chemical kinetics, the differential equations for the rates of the reactions are seldom used because the integrated expressions are much more accurate. The use of numerical integration of the calorimetric data also results in greater accuracy. Integrals can be numerically evaluated by using the mean value of the function, and the integration limits. Mathematically,

$$\int_a^b f(x)dx = (b-a) \langle f(x) \rangle \quad (\text{A.15})$$

where a and b are the integration limits and $\langle f(x) \rangle$ is the mean value of the function. Other approximate integration methods can also be used such as Simpson's Rule or the Trapezoidal Rule, but only A.15 is, strictly speaking, correct in that it agrees with the mathematical definition of an integral. The Trapezoidal Rule, however, allows integration around the discontinuities at $t=t_1$ and $t=t_2$. The integrals of power over selected time periods give units of energy (J).

The required heat capacities at constant pressure (C_p), enthalpy of vaporization of D₂O (L), the vapor pressure of D₂O (P), and the thermoneutral potential (E_H) are available from thermodynamic tables at 25°C (298.15K) and standard pressure. The temperature dependence of these calorimetric parameters at standard atmospheric pressure can be calculated from the following equations where T is the Kelvin temperature (°C + 273.15).

$$L = 85263.9 - 173.429T + 0.2586T^2 - 1.91913 \times 10^{-4} T^3 - 1805569T^{-1} \text{ (in J/mol}^1) \quad (\text{A.16})$$

$$E_H = 1.5318346 - 0.0002067(T - 273.15) \text{ (in V)} \quad (\text{A.17})$$

$$\log P = 35.47686 - 3343.93T^{-1} - 10.9 \log T + 0.0041645T + 9.14056/(197.397 - T) \quad (\text{P in atm}) \quad (\text{A.18})$$

$$C_{p,D_2O(l)} = 200.13 - 495.9 \times 10^{-3} T + 573.07 \times 10^{-6} T^2 - 16.765 \times 10^5 T^{-2} \text{ (in J mol}^1 K^{-1}) \quad (\text{A.19})$$

$$C_{p,D_2O(g)} = 26.7006 + 21.2897 \times 10^{-3} T - 2.66774 \times 10^{-6} T^2 + 1.2907 \times 10^5 T^{-2} \text{ (in J mol}^1 K^{-1}) \quad (\text{A.20})$$

$$C_{p,D_2(g)} = 28.9778 - 1.49226 \times 10^{-3} T + 4.14779 \times 10^{-6} T^2 + 0.26544 \times 10^5 T^{-2} \text{ (in J mol}^1 K^{-1}) \quad (\text{A.21})$$

$$C_{p,O_2(g)} = 23.1436 + 18.2628 \times 10^{-3} T - 6.605 \times 10^{-6} T^2 + 1.2118 \times 10^{-5} T^{-2}$$

(in $J mol^{-1} K^{-1}$)

(A.22)

At a typical cell temperature of 60°C (333.15 K) and standard pressure, these calculated values are $L = 43672 J mol^{-1}$, $E_H = 1.5194 V$, $P = 0.18317 atm$, $C_{p,D_2O(l)} = 83.420 J mol^{-1} K^{-1}$, $C_{p,D_2O(g)} = 34.660 J mol^{-1} K^{-1}$, $C_{p,D_2(g)} = 29.180 J mol^{-1} K^{-1}$, and $C_{p,O_2(g)} = 29.587 J mol^{-1} K^{-1}$.

Although the radiative and conductive heat transfer coefficients, k_R and k_C in Eqs. A.5 and A.6, are independent of the cell temperature, the use of the pseudoradiative heat transfer coefficient (Eq. 2) introduces a slight temperature dependence [2]. From Eq. 2,

$$P'_R = -[k_R(T_{cell}^4 - T_b^4) + k_C(T_{cell} - T_b) + RT_{cell}(0.75 I / F)] = -k'_R(T_{cell}^4 - T_b^4)$$
(A.23)

Solving Eq. A.23 for k'_R yields

$$k'_R = k_R + [k_C(T_{cell} - T_b) + RT_{cell}(0.75 I / F)] / (T_{cell}^4 - T_b^4)$$
(A.24)

As the cell temperature increases, k'_R decreases and approaches more closely the value of k_R . For Dewar cells with good vacuums, k'_R will be close to k_R [1, 2].

Another approach that has been used is to express the conductive heat transport contribution to k'_R by the use of $T_{cell} = T_b + \Delta T$ [1, 4]. Expansion by the binomial theorem yields

$$T_{cell}^4 - T_b^4 = (T_b + \Delta T)^4 - T_b^4 = 4T_b^3 \Delta T + 6T_b^2 \Delta T^2 + 4T_b \Delta T^3 + \Delta T^4$$
(A.25)

Each term in this expansion becomes smaller as the power of T_b decreases. The first term is the largest and is similar to a conductive term because it is proportional to ΔT . Therefore, the conductive heat transfer coefficient can also be expressed approximately by

$$k_C = 4 k_R \varphi T_b^3$$
(A.26)

where φ is the fraction of the heat transferred by conduction. By assuming $\varphi = 0.05$, $k_R = 0.8000 \times 10^{-9} WK^{-4}$, $T_b = 20^\circ C$ (293.15), $T_{cell} = 60^\circ C$ (333.15 K), and $I = 0.2000 A$, then from Eq. A.26, $k_C = 0.004031 WK^{-1}$, and from Eq. A.23, $P_R = -3.9467 W$, $P_C = -0.1612 W$, $P_W = -0.0043 W$, thus $P'_R = -4.1122 W$ and $k'_R = 0.8336 \times 10^{-9} WK^{-4}$. Therefore, for these assumptions using typical experimental conditions, the contributions of P_C and P_W to P'_R are only 3.92% and 0.10%, respectively.

It has been falsely asserted that conductive heat transfer pathways such as through the cell top or via the wire leads can cause large errors in this Dewar calorimetric cell. However, calculations using known thermal conductivities yield less than 1 mW for the rate of heat transfer by the wire

leads in this cell [2]. From Eq. A.23, it can be readily shown that the actual radiative power, P_R , is given by

$$P_R = -k_R (T_{cell}^4 - T_b^4) = P'_R (k_R / k'_R) \quad (\text{A.27})$$

Therefore, if only 50% of the heat transfer was by radiation ($P_R = 0.5 P'_R$), then $k'_R = 2 k_R$. For Dewar calorimetric cells with good vacuums, $k'_R \approx k_R$ as calculated by the Stefan – Boltzmann coefficient, thus the rate of heat transfer by conduction is much smaller than the rate of heat transfer by radiation.

APPENDIX II. List of Symbols

C_p	= heat capacity at constant pressure, JK ⁻¹ mol ⁻¹
E_H	= thermoneutral potential, V
F	= Faraday constant, 96485.3415 C mol ⁻¹
I	= cell current, A
k_C	= conductive heat transfer coefficient, WK ⁻¹
k'_C	= pseudoconductive heat transfer coefficient, WK ⁻¹
k_R	= radiative heat transfer coefficient, WK ⁻⁴
k'_R	= pseudoradiative heat transfer coefficient, WK ⁻⁴
L	= enthalpy of evaporation for D ₂ O, J mol ⁻¹
n_g	= moles of electrolysis gases, mol
M	= heavy water equivalent of the calorimetric cell, mol
P	= partial pressure of D ₂ O, Pa
P^*	= atmospheric pressure, Pa
P_C	= power transferred by conduction, W
P_{calor}	= rate of enthalpy change within the calorimeter, W
P_{EI}	= power input due to electrolysis, W
P_{gas}	= rate of enthalpy transport by the gas stream, W
P_H	= power input due to the calibration heater, W
P_R	= power transferred by radiation, W
P_W	= rate of pressure-volume work by the generated gases, W
P_X	= excess power generated, W
R	= gas constant, 8.314472 JK ⁻¹ mol ⁻¹
T_b	= temperature of water bath, K
T_{cell}	= temperature of cell, K
ΔT	= $T_{cell} - T_b$, K
$f(T)$	= $T_{cell}^4 - T_b^4$, K ⁴
α	= rate of change of k'_R , s ⁻¹
β	= fraction of D ₂ O lost by evaporation, dimensionless
γ	= current efficiency for electrolysis, dimensionless
φ	= fraction of heat transferred by conduction, dimensionless

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